AD

Technical Report ARWSB-TR-17026

Initial Assessment of CSA Group Niobium-Boron Based Coatings on 4340 Steel

C.P. Mulligan

July 2017

ARMAMENT RESEARCH, DEVELOPMENT AND ENGINEERING CENTER
Weapons Software & Engineering Center
Benét Laboratories
Watervliet, NY 12189

REPORT DOCUMENTATION PAGE	*Form Approved* **OMB No. 0704-0188**

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED** *(From - To)*
July 2017	Technical	

4. TITLE AND SUBTITLE Initial Assessment of CSA Group Niobium-Boron Based Coatings on 4340 Steel	**5a. CONTRACT NUMBER** **5b. GRANT NUMBER** **5c. PROGRAM ELEMENT NUMBER**
6. AUTHOR(S) C.P. Mulligan	**5d. PROJECT NUMBER** **5e. TASK NUMBER** **5f. WORK UNIT NUMBER**
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army ARDEC Benet Laboratories, RDAR-WSB Watervliet, NY 12189-4000	**8. PERFORMING ORGANIZATION REPORT NUMBER** ARWSB-TR-17026
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	**10. SPONSOR/MONITOR'S ACRONYM(S)** **11. SPONSORING/MONITORING AGENCY REPORT NUMBER**

12. DISTRIBUTION AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT
Three (3) samples were received from CSA Group for initial assessment of coating properties. The specimens were arbitrarily labeled as CSA-1, CSA-2, and CSA-3. The vendor also supplied two (2) metallographic mounts reported as (1) thin and (2) thick Niobium-Boron (Nb-B) type coatings on steel. CSA Group is interested in providing coatings for potential electroplated Cr replacement. The group reportedly has the capability to deposit refractory metals on steel. The Army has interest in Cr replacement technologies both for performance and environmental considerations. This initial assessment is intended to determine applicability of the coatings for protection of high-strength steel.

15. SUBJECT TERMS
Niobium-Boron Based Coatings; 4340 Steel; CSA Group

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**	U	16	C.P. Mulligan
U/U	U	U			**19b. TELEPONE NUMBER (*Include area code*)** (518) 266-5415

INSTRUCTIONS FOR COMPLETING SF 298

1. REPORT DATE. Full publication date, including day, month, if available. Must cite at lest the year and be Year 2000 compliant, e.g., 30-06-1998; xx-08-1998; xx-xx-1998.

2. REPORT TYPE. State the type of report, such as final, technical, interim, memorandum, master's thesis, progress, quarterly, research, special, group study, etc.

3. DATES COVERED. Indicate the time during which the work was performed and the report was written, e.g., Jun 1997 - Jun 1998; 1-10 Jun 1996; May - Nov 1998; Nov 1998.

4. TITLE. Enter title and subtitle with volume number and part number, if applicable. On classified documents, enter the title classification in parentheses.

5a. CONTRACT NUMBER. Enter all contract numbers as they appear in the report, e.g. F33615-86-C-5169.

5b. GRANT NUMBER. Enter all grant numbers as they appear in the report, e.g. 1F665702D1257.

5c. PROGRAM ELEMENT NUMBER. Enter all program element numbers as they appear in the report, e.g. AFOSR-82-1234.

5d. PROJECT NUMBER. Enter al project numbers as they appear in the report, e.g. 1F665702D1257; ILIR.

5e. TASK NUMBER. Enter all task numbers as they appear in the report, e.g. 05; RF0330201; T4112.

5f. WORK UNIT NUMBER. Enter all work unit numbers as they appear in the report, e.g. 001; AFAPL30480105.

6. AUTHOR(S). Enter name(s) of person(s) responsible for writing the report, performing the research, or credited with the content of the report. The form of entry is the last name, first name, middle initial, and additional qualifiers separated by commas, e.g. Smith, Richard, Jr.

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES). Self-explanatory.

8. PERFORMING ORGANIZATION REPORT NUMBER. Enter all unique alphanumeric report numbers assigned by the performing organization, e.g. BRL-1234; AFWL-TR-85-4017-Vol-21-PT-2.

9. SPONSORING/MONITORS AGENCY NAME(S) AND ADDRESS(ES). Enter the name and address of the organization(s) financially responsible for and monitoring the work.

10. SPONSOR/MONITOR'S ACRONYM(S). Enter, if available, e.g. BRL, ARDEC, NADC.

11. SPONSOR/MONITOR'S REPORT NUMBER(S). Enter report number as assigned by the sponsoring/ monitoring agency, if available, e.g. BRL-TR-829; -215.

12. DISTRIBUTION/AVAILABILITY STATEMENT. Use agency-mandated availability statements to indicate the public availability or distribution limitations of the report. If additional limitations/restrictions or special markings are indicated, follow agency authorization procedures, e.g. RD/FRD, PROPIN, ITAR, etc. Include copyright information.

13. SUPPLEMENTARY NOTES. Enter information not included elsewhere such as: prepared in cooperation with; translation of; report supersedes; old edition number, etc.

14. ABSTRACT. A brief (approximately 200 words) factual summary of the most significant information.

15. SUBJECT TERMS. Key words or phrases identifying major concepts in the report.

16. SECURITY CLASSIFICATION. Enter security classification in accordance with security classification regulations, e.g. U, C, S, etc. If this form contains classified information, stamp classification level on the top and bottom of this page.

17. LIMITATION OF ABSTRACT. This block must be completed to assign a distribution limitation to the abstract. Enter UU (Unclassified Unlimited) or SAR (Same as Report). An entry in this block is necessary if the abstract is to be limited.

ABSTRACT

Three (3) samples were received from CSA Group for initial assessment of coating properties. The specimens were arbitrarily labeled as CSA-1, CSA-2, and CSA-3. The vendor also supplied two (2) metallographic mounts reported as (1) thin and (2) thick Niobium-Boron (Nb-B) type coatings on steel. CSA Group is interested in providing coatings for potential electroplated Cr replacement. The group reportedly has the capability to deposit refractory metals on steel. The Army has interest in Cr replacement technologies both for performance and environmental considerations. This initial assessment is intended to determine applicability of the coatings for protection of high-strength steel.

Table of Contents

List of Figures

List of Tables

1. BACKGROUND

CSA Group is interested in providing coatings for potential electroplated Cr replacement. The group reportedly has the capability to deposit refractory metals on steel. The Army has interest in Cr replacement technologies both for performance and environmental considerations.

2. EXPERIMENTAL PROCEDURE

2.1. Coating Specimens

Three (3) each 3”x5” 4340 plates in the quenched and tempered condition (HRC 35-43) were sent to CSA Group for coating. Prior teleconference indicated that CSA Group has the capability to coat specimens with Nb, Ta, and Cr. Deposition temperature was reported to be ~1350 °F. All coatings received were of the Niobium-Boron (Nb-B) type.

2.2. Coating Thickness

Coating thickness measurements were made with a magnetic lift-off gauge (Karl Deutsch Model Leptoskop 2040) on each coated plate. A minimum of five (5) measurements were taken on each side of each plate.

2.3. ASTM Adhesion Testing

Adhesion testing was conducted in accordance with ASTM B571, “Standard Practice for Qualitative Adhesion Testing of Metallic Coatings”, using Method 13.1 – "Scribe-Grid Test”. In this test a rectangular grid pattern is scribed and if any portion of the coating between the lines exhibits adhesive failure, the adhesion is deemed inadequate. The test is particularly aggressive for hard coatings.

2.4. Microstructural/Composition Evaluation

Specimens were prepared in metallographic mounts for cross-sectional evaluation. Both adhesion test samples as well as samples in metallographic mounts were examined in an FEI Nanolab 600i Field Emission Scanning Electron Microscope (FE-SEM) operating at 10 kV and equipped with Energy Dispersive Spectroscopy (EDS) for elemental analysis.

3. RESULTS

3.1. Coating Specimens As-Received

Three (3) samples were received for evaluation, arbitrarily labeled as CSA-1, CSA-2, and CSA-3. In addition, the vendor supplied two (2) metallographic mounts reported as (1) thin and (2) thick Niobium-Boron (Nb-B) type coatings on steel. Representative Images of each of the coated plate specimens for are given Figure 1. The surfaces have an irregular appearance. CSA-1 exhibited a blackened rough surface on one side of the specimen.

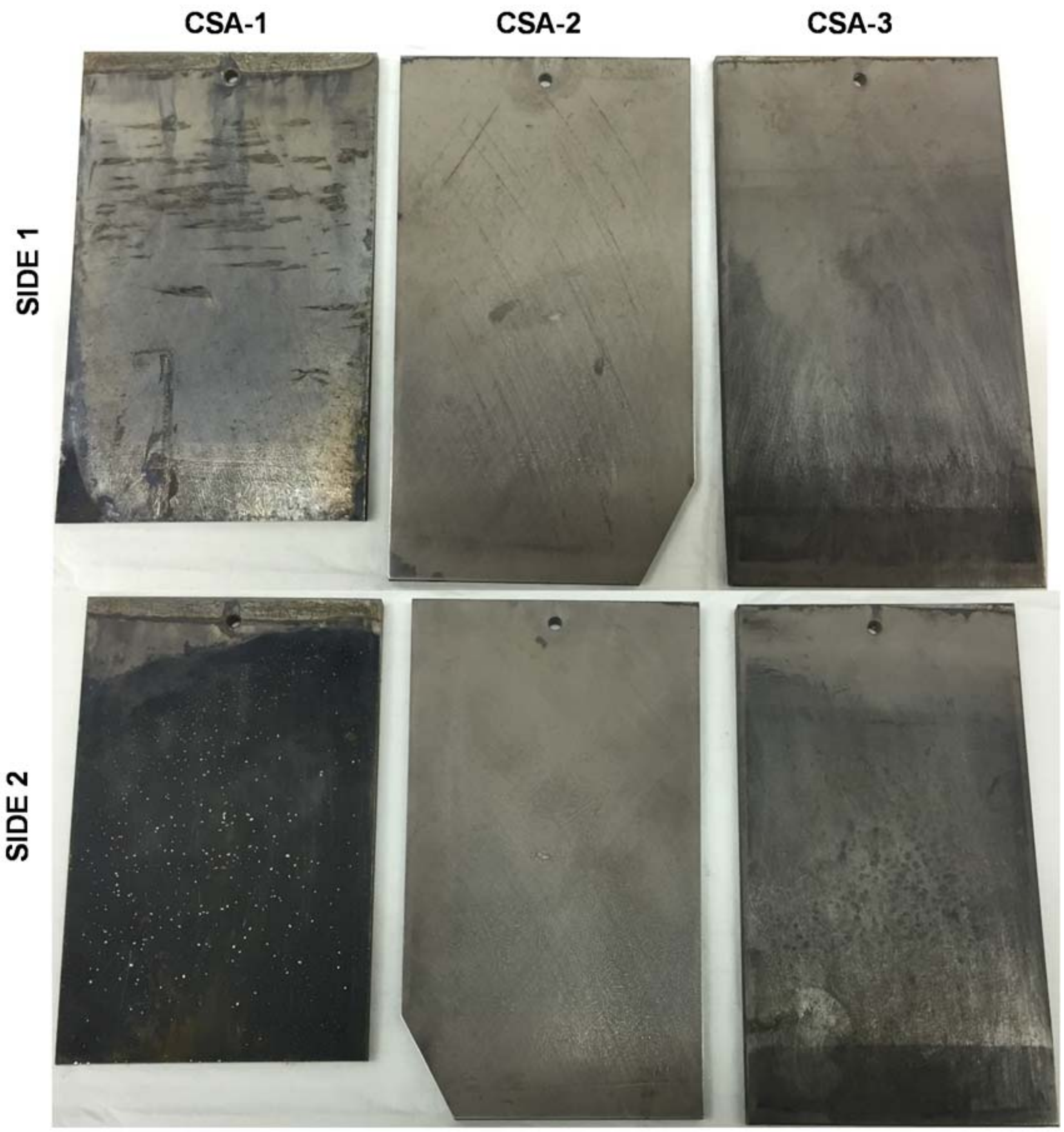

Figure 1: As-coated plate specimens.

3.2. Coating Thickness Measurements

Table I gives the measured coating thickness for both sides of all as-received specimens. CSA-1 was the thickest coating overall. All coatings were in the range of 0.0002-0.0015” (0.2-1.5 mil). This is significantly thinner than the typical engineering Cr plate thickness of ~0.005”. The vendor reports that coating thickness of up to 0.002” are achievable for the Nb-B based coating.

Table I: Coating thickness data for as-received specimens (data in mils).

Coating thickness in mils (0.001")						
	CSA-1		CSA-2		CSA-3	
Measurement	SIDE 1	SIDE 2	SIDE 1	SIDE 2	SIDE 1	SIDE 2
1	0.24	1.30	0.04	0.35	0.16	0.20
2	0.28	0.71	0.04	0.31	0.12	0.16
3	0.67	1.46	0.08	0.12	0.12	0.12
4	0.35	1.46	0.12	0.12	0.16	0.24
5	0.28	1.10	0.16	0.20	0.12	0.24
AVG	0.36	1.20	0.09	0.22	0.13	0.19
STDEV	0.18	0.31	0.05	0.11	0.02	0.05

3.3. Adhesion Performance

Adhesion testing was completed on 1”x1” specimens cut from each plate. SEM/EDS evaluation indicates that all the coatings were the Nb-B type coatings and that no adhesive failure as observed. Cohesive brittle fracture of the coating was observed which is typical of hard coatings. This indicates the coatings have excellent adhesion. Figures 2 and 3 illustrate the surface images of the scribe-grid pattern along with EDS results showing no exposed steel. However, the coating does appear to have a multi-layered type structure with the top layer having a significantly higher oxygen concentration indicating some level of oxidation of the surface. The surface itself is nodular in appearance. Boron was observed to be present for each of the coatings. Figures 4 and 5 give the similar images for CSA-2 and CSA-3.

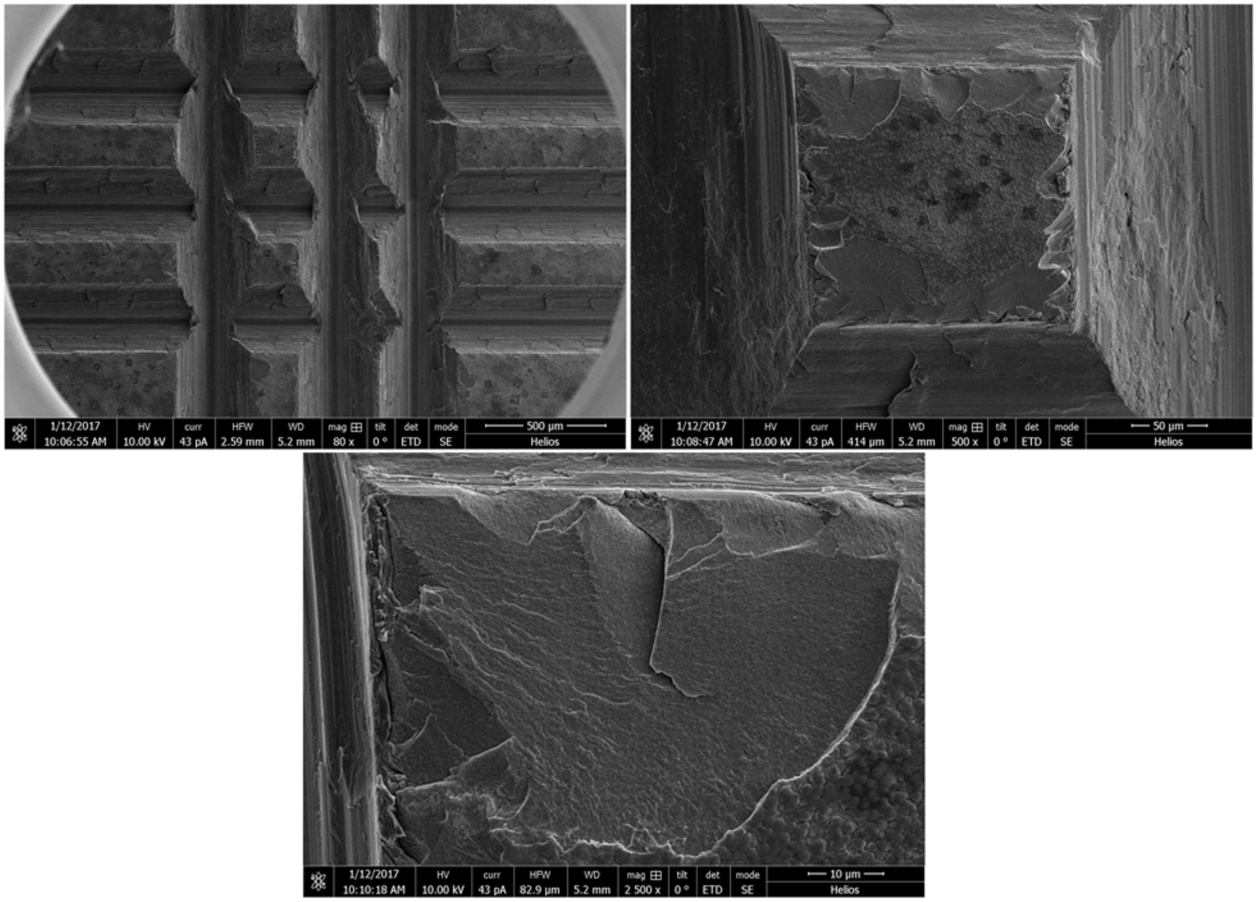

Figure 2: SEM micrographs of CSA-1 subjected to adhesion testing.

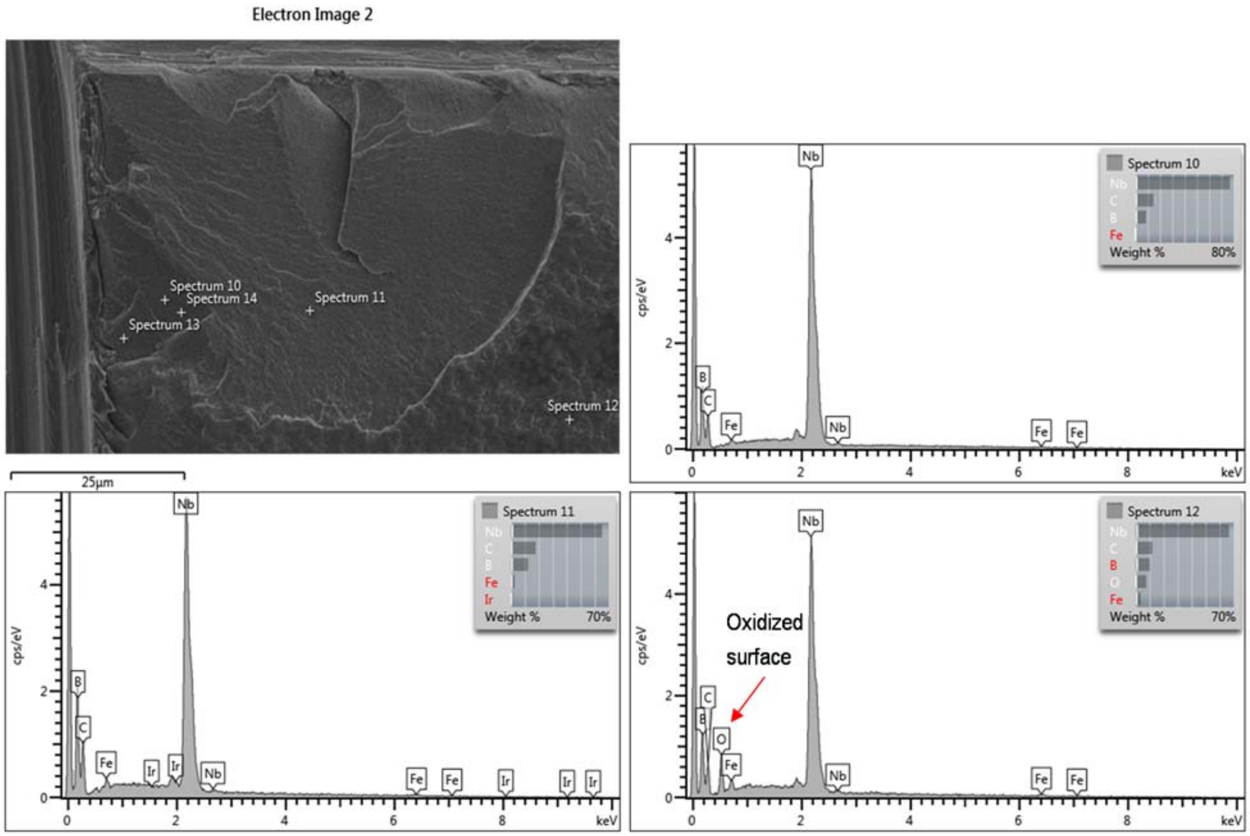

Figure 3: SEM micrograph with EDS spectra indicating no exposed steel for CSA-1.

Distribution A: Approved for public release; distribution unlimited.

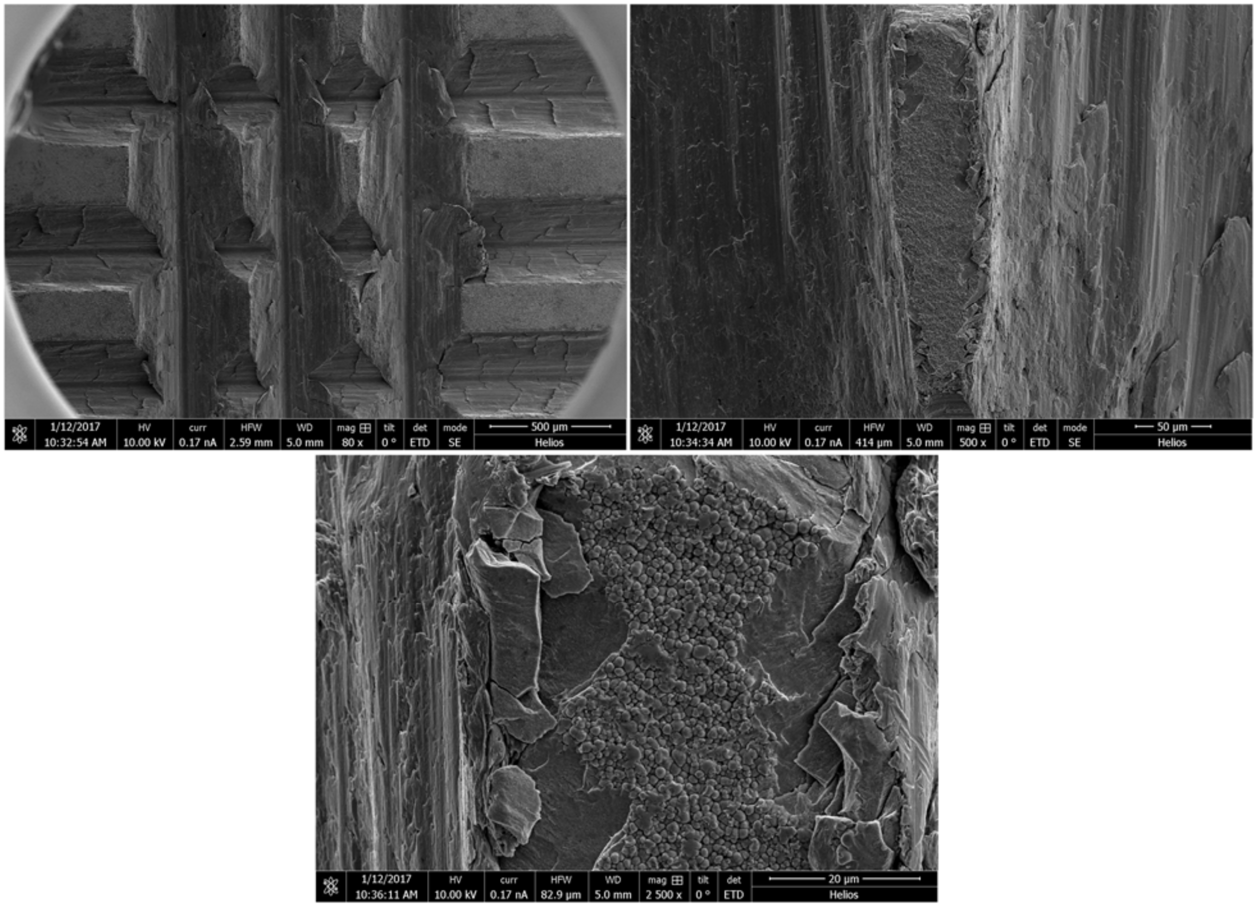

Figure 4: SEM micrographs of CSA-2 subjected to adhesion testing.

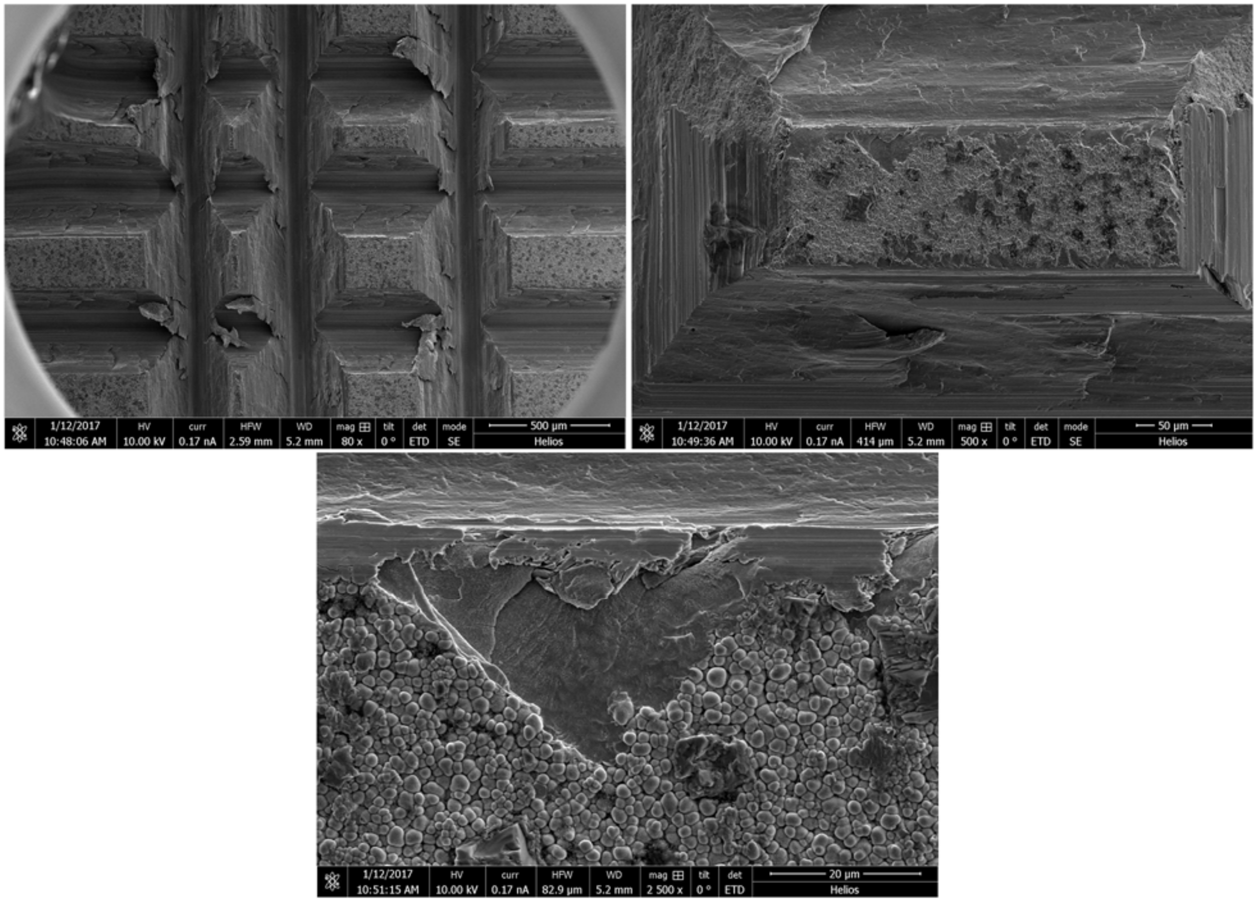

Figure 5: SEM micrographs of CSA-3 subjected to adhesion testing.

3.4. Microstructure/Composition

Metallographic examination was completed on each of the specimens. Figure 6-8 illustrate cross-sections of all the coatings with images at 500X magnification following a steel Nital etch. Figure 6 shows CSA-1 Side 1 and Side 2 coating. Figures 7 and 8 show the thickest sides for CSA-2 and CSA-3 respectively along with views of the steel microstructure. The microstructures of the specimens indicate various mixes of martensite, bainite, and retained austenite. This indicates that the austenitizing temperature of the steel of ~1375 °F was exceeded either during the coating process or in a post-processing step. Figure 9 illustrates an image of the “thick” Nb-B coated metallographic specimen provided by CSA Group indicating that a thickness of 0.002” is achievable.

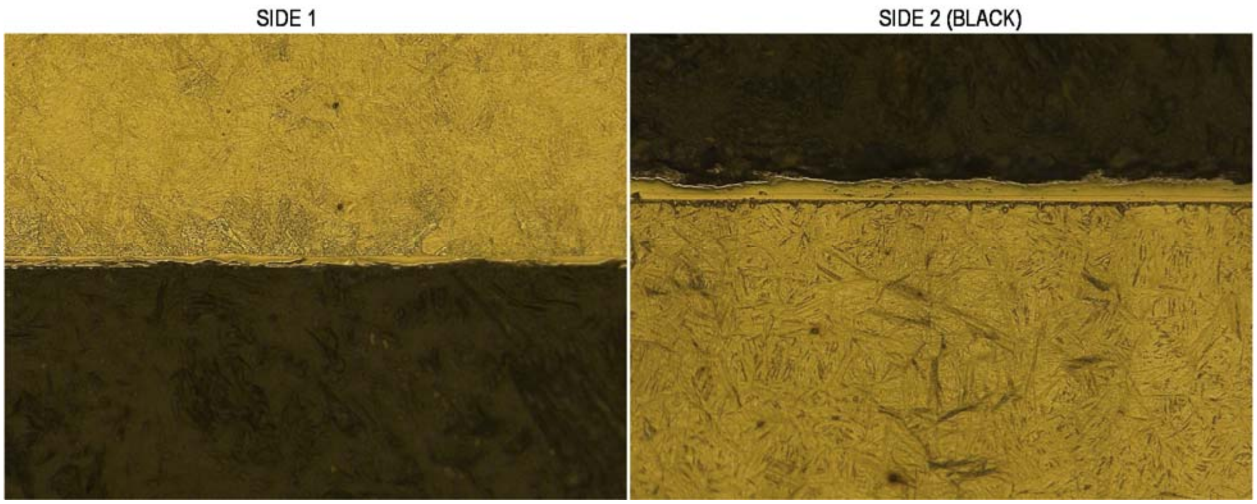

Figure 6: Metallographic images of CSA-1 specimen.

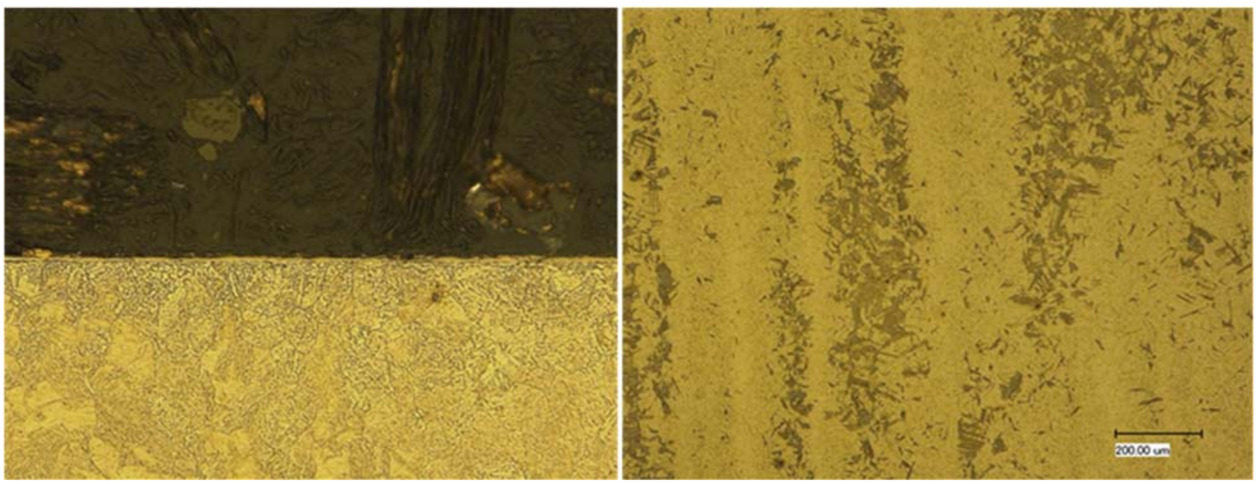

Figure 7: Metallographic images of CSA-2 specimen.

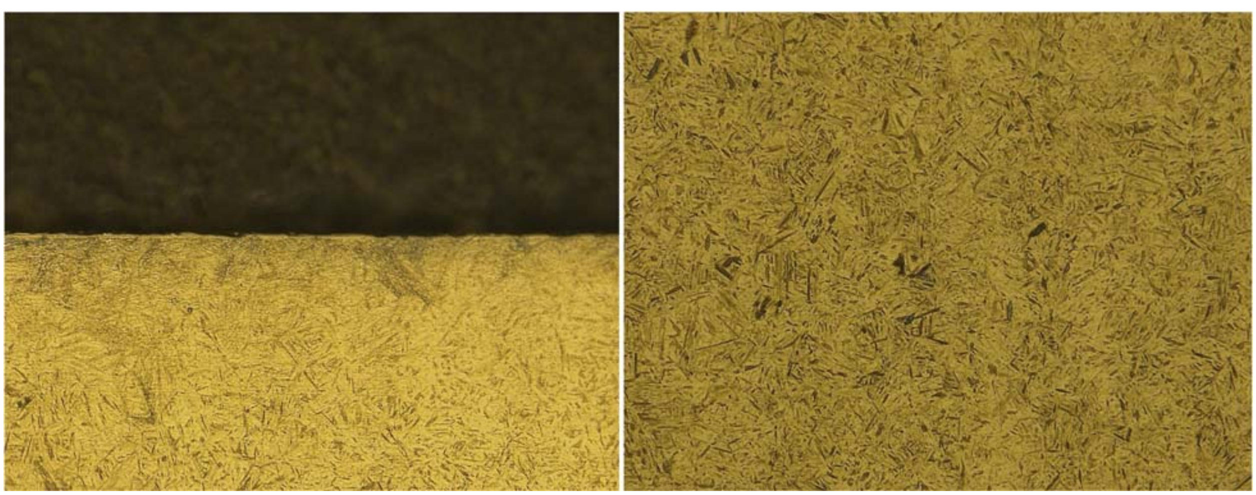

Figure 8: Metallographic images of CSA-3 specimen.

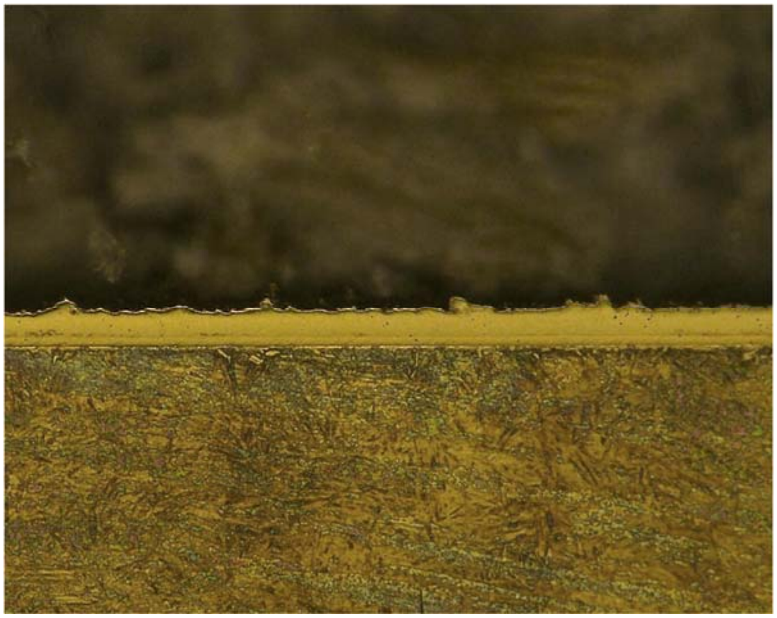

Figure 9: Metallographic image of CSA Group provided metallographic specimen.

Figure 10 illustrates an SEM micrograph and associated EDS spectra for the CSA-2 specimen. The coating exhibits a duplex type structure with a layer of higher oxygen content at the surface as illustrated by spectrum 1. Spectrum 2 indicates a Nb rich layer while spectrums 3, 4, and 5 show the steel substrate.

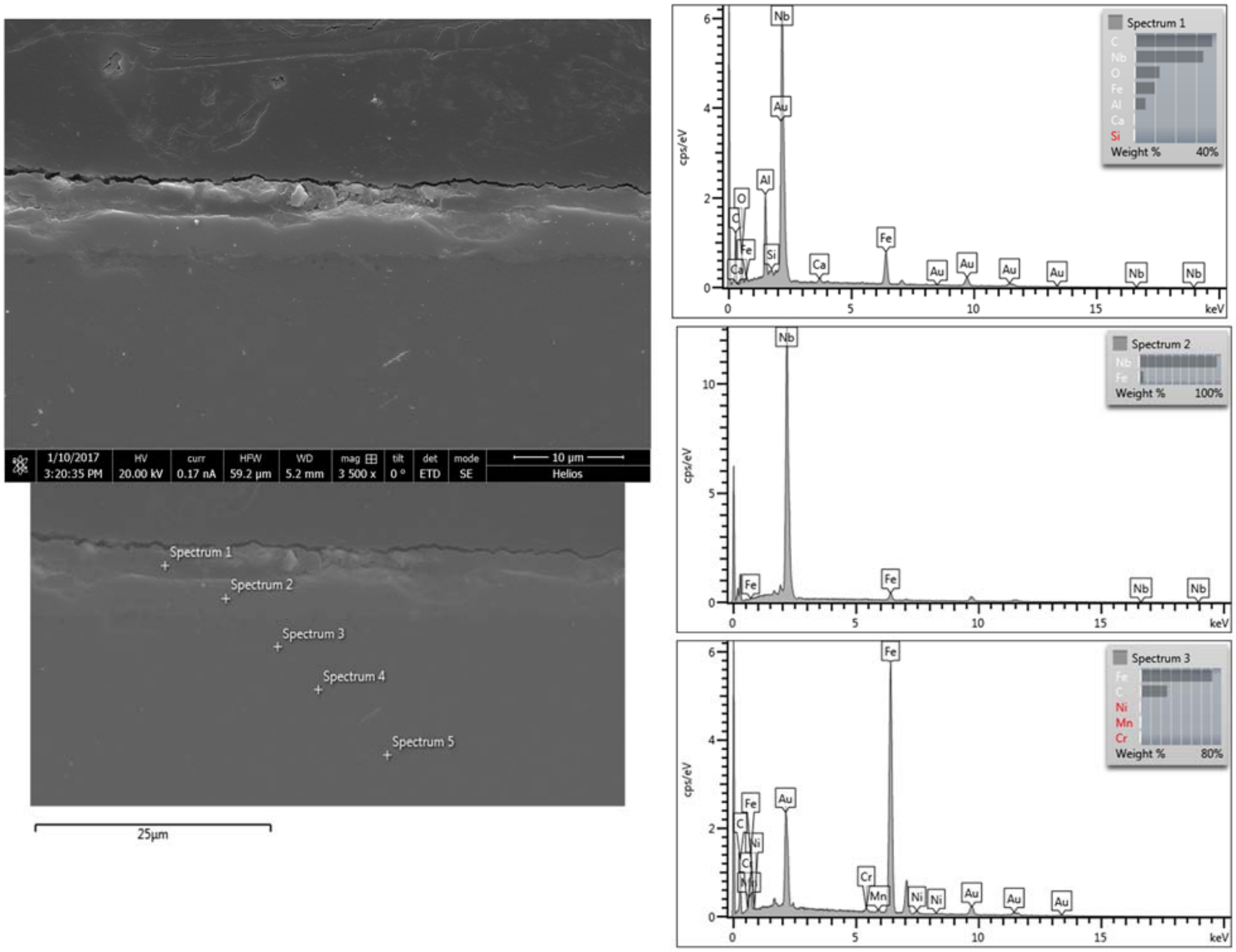

Figure 10: SEM micrograph and associated EDS spectra for cross-section of CSA-2 (Note: metallographic specimen was coated with gold to facilitate evaluation which interferes with the Nb peak).

Figure 11 illustrates surface images of the CSA-1 specimen. This is for the non-blackened Side 1 of the specimen. Figure 12 gives a micrograph and associated EDS spectra indicating the dark areas are regions of oxidized material. Figures 13 thru 15 provide similar information for CSA-2 and CSA-3. An interesting feature observed on the surface of CSA-3 is a network of nanowires emanating from some of the surface grains of the coating.

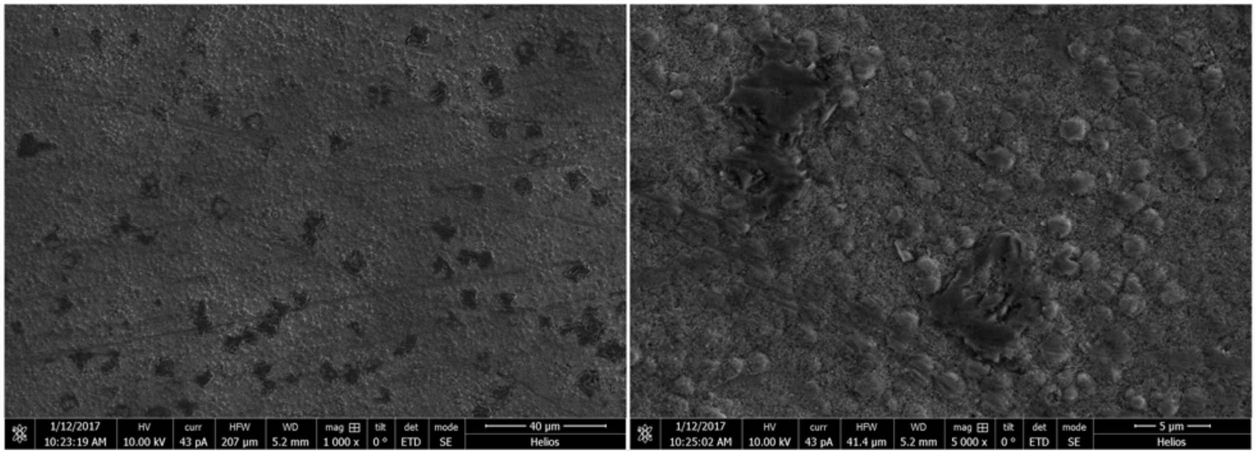

Figure 11: SEM micrographs of surface of CSA-1.

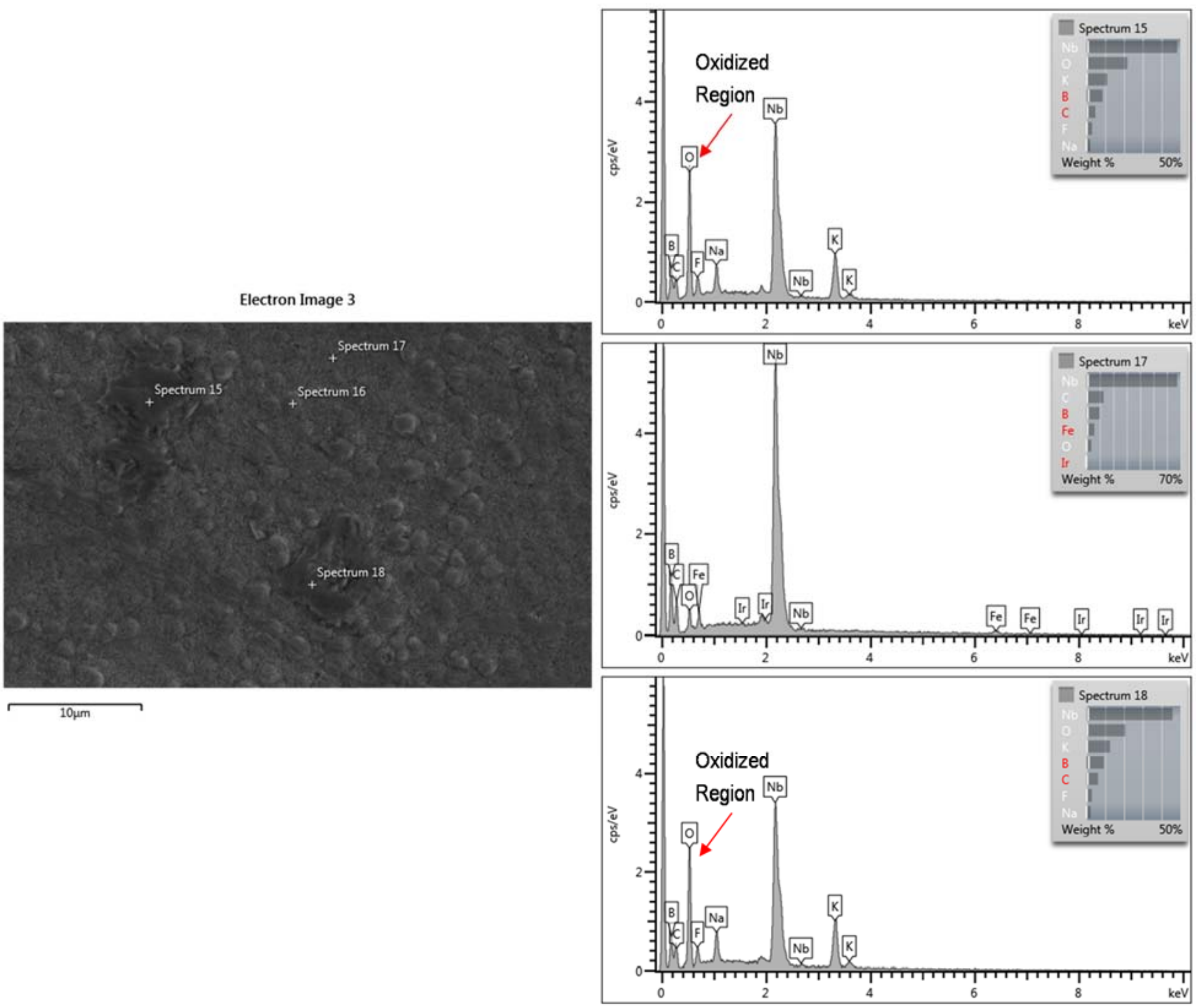

Figure 12: SEM micrograph and associated EDS spectra for CSA-1 surface.

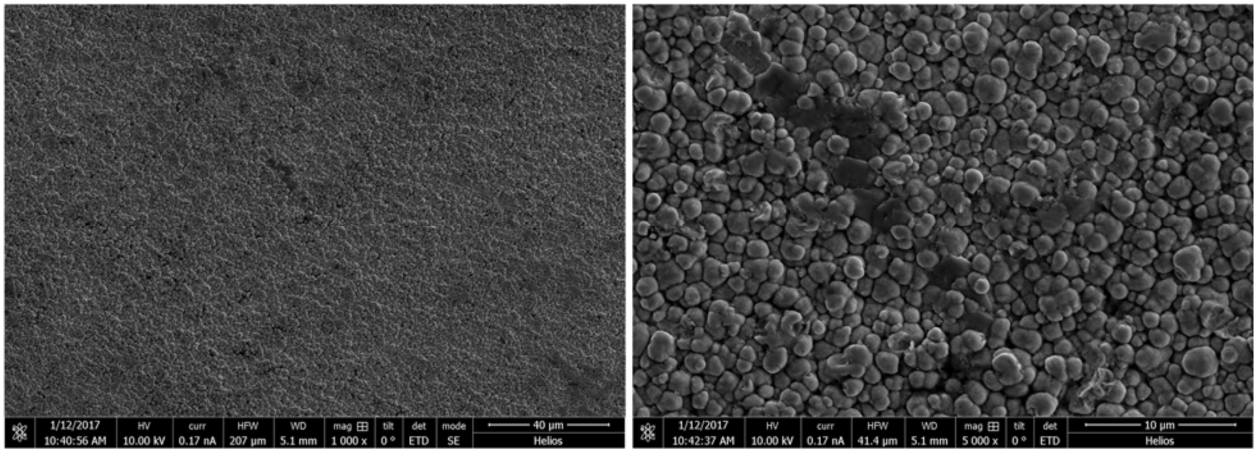

Figure 13: SEM micrographs of surface of CSA-2.

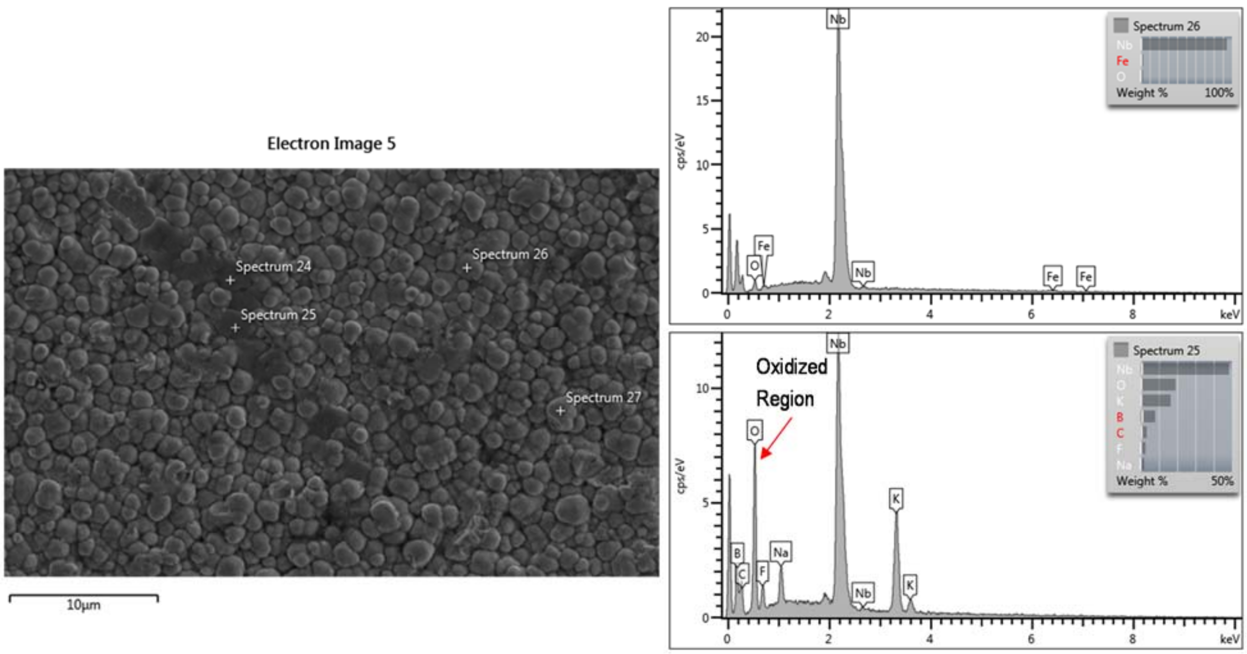

Figure 14: SEM micrograph and associated EDS spectra for CSA-2 surface.

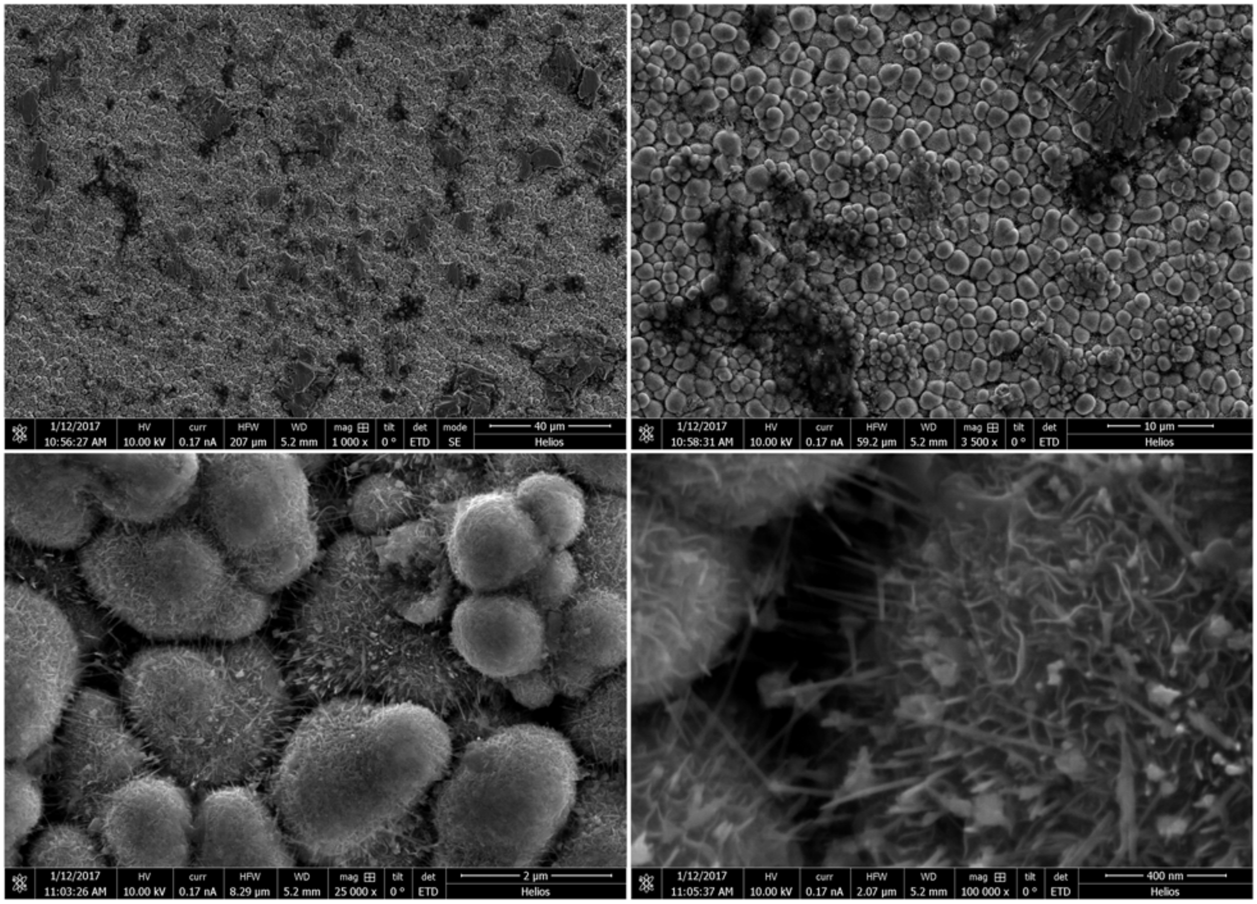

Figure 15: SEM micrographs of surface of CSA-3.

4. DISCUSSION AND CONCLUSIONS

CSA Group has successfully demonstrated a well-adhered Nb-B based layer on 4340 steel. The material itself could prove useful for a number of applications. However, there are several issues with respect to processing temperatures that pose issues for high strength steels. Evaluation indicates a temperature higher than 1375 °F (4340 austenitization temperature) was reached at some point in the processing of the specimens based on the characteristics of the steel microstructure. This is undesirable as it would necessitate a post coating heat treatment in order to bring back strength and ductility to the steel. Typically coating processes must be kept below the tempering temperature of the steel (typically in the range of 800-1200 °F) to maintain strength. In the case where residual stresses are introduced for the purpose of fatigue life extension of high strength steel components, the allowable temperatures are typically even lower. The high level of surface oxidation also indicates that the surface was exposed to an oxidizing atmosphere at elevated temperature. It is unclear if this occurred during the coating process or during some type of thermal anneal following coating to promote diffusion.

ACKNOWLEDGEMENTS

This initial assessment was completed with financial support from the SERDP Office.